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CONTRACT NO: DA-18-108-CML-5839

MONTHLY REPORT NO: 6

PERIOD: MARCH 1 - 31, 1956

PITTSBURGH COKE & CHEMICAL COMPANY

DATE: April 26, 1956

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PITTSBURGH COKE & CHEMICAL COMPANY

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ABSTRACT:

ASC Whetlerite:

The results this month are of very doubtful value. We had not taken the precaution of purifying the air used during equilibration since the compressor intake was in a non-vapor contaminated area in a room away from any other operations. During much of the equilibration in this period the compressor room was being painted and, unfortunately no one thought about carbon contamination until an explanation for unexpected results was necessary. We are reporting the results obtained, but feel that the only conclusion is that organic vapors make all the ageing materials give equivalent results. This in itself is rather interesting, if we knew what to make of it.

Mustard:

Calculations were made on the amount of mustard adsorbed to get a monomolecular layer on PCC CWS carbon and then the amounts adsorbed assuming no adsorption for pores below designated diameters. This would give us an idea as to what effect impregnants could have on desorption by occupying pores and, secondly what part of any mustard loading might be expected to be in outer pores. The mustard molecule was assumed to be cubic and have normal liquid density. Using available area from 10 Å pores and up 67 g. of H/100 g. of C would give a monofilm, from 16 Å pores and up 53 g., 20 Å and up 14 g. of H, 30 Å and up 3 g. of H, etc. We have studied desorption at H loadings from about 5 to 50 g./100 g. of C. As an approximation complete void or pore filling would be around 110 g. of H/100 g. of C.

A number of impregnations and treatments were given carbon and mustard desorption measured from them. About the same results as have been previously reported resulted. Impregnants generally increase the desorption rate. Treatments with aqueous acid solutions were generally beneficial, however. The results with non-volatile acids where acid was retained by the carbon, i.e., H_2SO_4 and H_3PO_4 gave only moderate to no effect. The volatile acids HCl, HNO_3, CH_3COOH gave definite improvement in most cases. In the latter case no acid was retained as measured by weight change of the carbon. Acids

absorbed in the pore structure probably balance the surface effect by occupying space otherwise available for mustard. Of course, we are measuring rate of reaction more than equilibrium so diffusion from inner pores would presumably always show reduced rates by our method.

ASC Whetlerite, V_2O_5, $Hg(CN)_2$, $HgBr_2$, MoO_3 all showed increased desorption as did ethanolamine and sulfamic acid. Of the volatile acids, HNO_3 showed the least desorption after a long time. If any nitric acid were retained it would oxidize mustard. The carbon ash nitrates might also oxidize mustard. Since the initial rate was the same as HCl and acetic acid there would be no advantage in the use of nitric acid.

On the possibility that acid washing removes ash which increased desorption, strong HCl solution was used several times on carbon. No increased benefit was obtained.

CONCLUSIONS:

1. Several inorganic salts or oxides had the same general effect as organic materials in increasing the desorption rate of mustard from carbon.

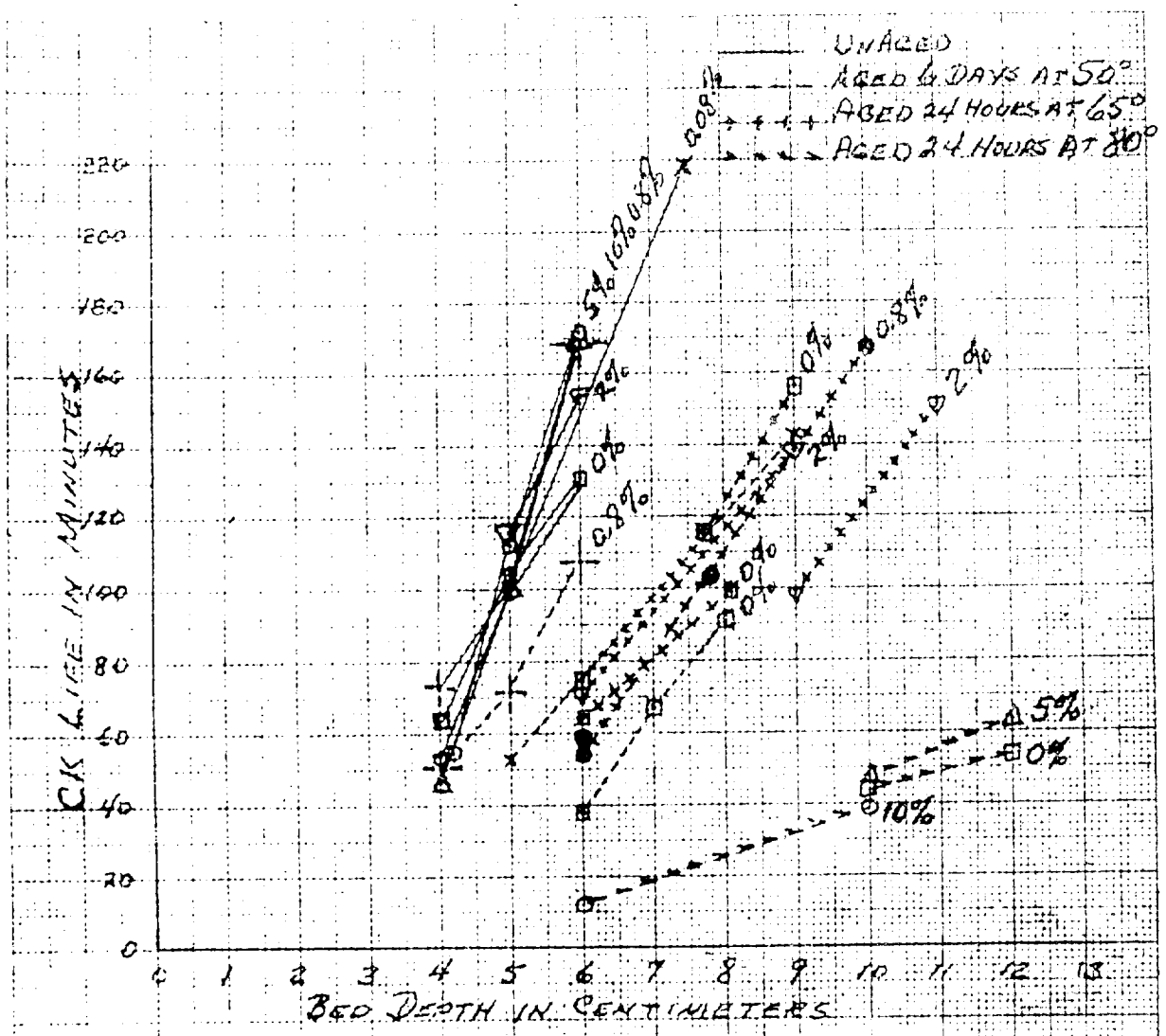
2. Treatment of carbon with volatile acids decreases the desorption rate, while treatment with non-volatile acids have only minor effects.

RESULTS:

ASC Whetlerite:

The whetlerites tested this month were described last month. Equilibration, ageing and testing were done under standard test conditions already described. Equilibration at 80% RH to constant weight takes around 20 + hours, so we normally run for 44 to 48 hours. Air flow is about 3.5 l./min. and thus around 10,000 liters of air are used. During the period of last month the room containing the compressor and the laboratory was being painted. The effect of organic vapors on carbon is obvious and we have always been careful in make-up and drying to avoid any contamination. CK tests are run in an area where no organic work is done and it was felt unnecessary to treat the air. For some reason the paint solvent problem did not occur to us until after the tests. As can be seen in Figure 1 and Table I, results indicate no advantage of soda ash treatment as did earlier data. However, ageing at 50°C. was more severe than usual. At present we are assuming that adsorbed organic vapors have changed the picture and are making no conclusions.

Chromate analysis are not complete in this series. Those available are reported in Table II. We hesitate to draw conclusions from them but there is an indication that Cr^{+6} reduction during ageing is not the reason for the results on CK life and that another effect such as the paint solvent adsorption is responsible for our results.



CK LIFES OF SOME LABORATORY IMPREGNATIONS OF PCuC CARBON

□ ASC STANDARD SOLUTION
ASCN-1 " " PLUS 0.8% NA_2CO_3
× ASCN-2 " " " 0.08% "
△ ASCN-3 " " " 5.0% "
▽ ASCN-4 " " " 2.0% "
● ASCN-6 " " " 6.0% "
○ ASCN-7 " " " 3.2% NAOH

Fig. 1

TABLE I

CK TUBE TESTS

Sample		Bed Depth cm.	Mg. CK	Equil. Weight g.	Corrected Life min.	Mg. CK per g.	Net Weight Change, m
ASCN-4 (Standard Solution plus 2% Na_2CO_3) Initial Life	•	5.0	721	9.5343	115.5	75.6	- 93.8
	•	6.0	958	11.4325	153.5	83.8	- 62.8
Aged 24 hours at 65°C.	•	6.0	445	11.4176	71.3	39.0	+ 69.8
	•	9.0	870	17.1239	139.5	50.7	+ 88.8
	•	9.0	614	16.9921	98.3	36.1	+158.9
	•	11.0	944	20.7683	151.2	45.5	+250.3
Aged 24.5 hours at 65°C.		6.0	338	11.5377	54.1	29.2	+101.1
ASC-5 (Standard Solution)	•	6.0	463	11.2594	74.1	41.1	+ 53.3
Aged 24 hours at 65°C.	•	7.7	721	14.4498	115.4	49.9	+ 84.3
	•	9.0	977	16.8823	156.6	57.8	+100.5
Aged 24.5 hours at 65°C.		6.0	401	11.4238	64.1	35.1	-
		8.1	623	15.3756	99.6	40.5	-
ASCN-6 (Standard Solution plus	•	6.0	328	11.2724	52.6	28.1	+ 80.8
0.8% Na_2CO_3) Aged 24 hours at 65°C.	•	10.0	1048	18.8854	167.7	55.4	+225.6
Aged 24.5 hours at 65°C.		6.0	366	11.6843	58.5	31.4	-
		7.8	638	15.1243	102.4	42.4	-
Aged 6 days at 50°C.	•	6.0	284	11.3736	45.5	25.0	-
ASCN-7 (Standard Solution plus 8.2% NaOH) Aged 6 days at 50°C.	•	6.0	250	11.9796	40.0	20.9	+ 33.4

• Designates samples equilibrated while painters were in building.

Above tests run at 80-80 RH, 1.56 lpm, 4.00 mg. CK per liter

Tube cross section = 2.77 sq. cm.

TABLE II

CHROMATE ANALYSIS

	% Na_2CO_3	% CrO_3 Unequilibrated	% CrO_3 Aged 6 days 50° AR	% CrO_3 Aged 6 days 50° Dry	% CrO_3 Aged 24 hours 65° AR	% CrO_3 Aged 24 hours 65° Dry	% CrO_3 Aged 24 hours 80° AR	% CrO_3 Aged 24 hours 80° Dry
ASC-5	0	2.22						
ASCN-6	0.8	2.08	1.53	1.74				
ASCN-4	2	2.05	1.29	1.46	1.64			
					1.30 (24.5 hrs.)	1.57		
ASCN-3	5	1.95					0.56	0.88
ASCN-7	10	3.08	1.78	1.88			0.62	0.86

The whetlerites were leached by shaking three hours in 100 ml. 7N NH_4OH. Dilutions were made and optical density read on spectrophotometer. The results were corrected to a dry weight basis by drying duplicate samples and determining amounts of moisture present.

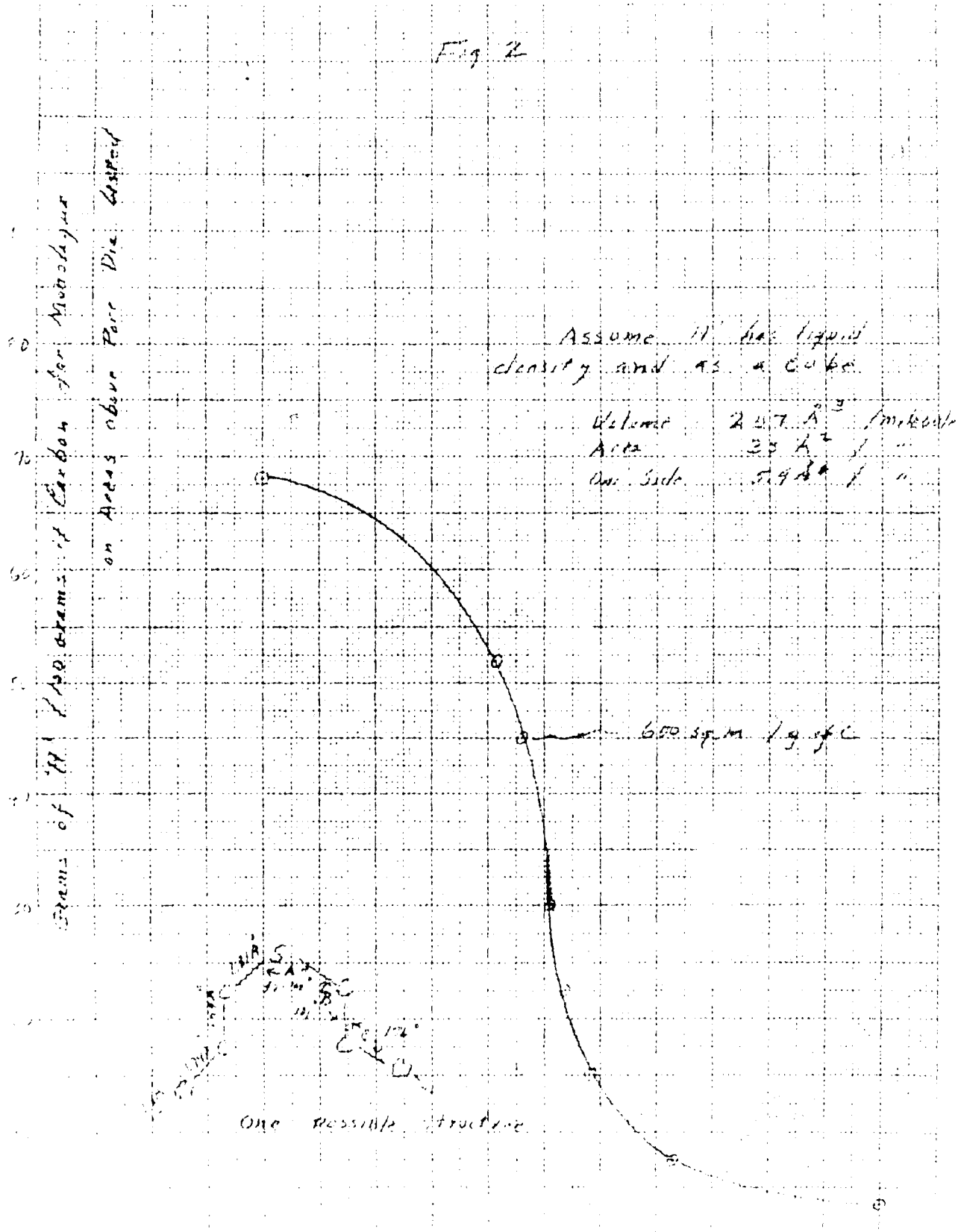
Results are expressed as percent CrO_3.

Mustard:

For purposes of general information calculations were made to find the amount of mustard adsorbed on CWS carbon assuming a monomolecular film on the surface. The carbon surface area was taken from the pore diameter, cumulative surface area curve determined from the water adsorption isotherm. Thus the amount of H adsorbed from the area available on pores above 10 Å, 15 Å, 20 Å diameter was determined and the results are plotted in Figure 2.

The volume of one molecule of mustard assuming normal liquid density is 207 cubic Ångstroms. Assuming cubic form the area of one face is 35 $Å^2$ and the side is 5.9 Å. Since 67 g. of H as a monolayer can be picked up by 100 g. of carbon on pores above 10 Å diameter, it should be possible to make mustard adsorption irreversible at room temperature by surface treatment of the carbon. However, pore plugging would have to be avoided and the preferred situation would be alteration of atoms only. For example, removing any polar bonds so that the ethylene group of the H would be held by C.

Fig 2



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MUSTARD:

Procedure:

A. The samples for this month, consisting of inorganic impregnants, acid dips, acid washes and organic impregnants were prepared following the procedure outlined in Monthly Report No. 5. This procedure should be corrected in that the samples were dried overnight rather than for three hours.

The adsorption of mustard was completed by placing the carbon treated sample in flat glass containers and then in a desiccator over mustard.

B. The rate of water desorption of mustard was determined following the procedures reported in Monthly Report No. 4.

Results:
A & B

The following tables summarize the results of the desorption runs using various loadings and dipping solutions. The figures are graphical interpretations of the same tables. The reference lines for ordinary CWS carbon at 10% and 20% mustard loading were taken from Monthly Report No. 5, Figure 4.

Due to an error in selection of carbon, samples of whetlerite were impregnated with various concentrations of thiodiglycol. Upon placing these samples in an oven for drying, it was found that a reaction had taken place for, the carbon was completely ashed at a temperature below 105°C.

TABLE III

DESORPTION OF MUSTARD FROM CARBON (WHETLERITE)

Whetlerite - 2.0000 g.
"H" - 0.4057 g. H/C = 20.3%
H_2O - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	60	25.8	6.4
2	70	25.4	6.3
3	130	39.9	9.3
4	205	52.9	13.0
5	265	60.7	15.0
6	335	69.0	17.0
7	390	74.2	18.3
8	450	77.1	19.0
9	1515	102.6	25.3

TABLE IV

DESORPTION OF MUSTARD FROM CARBON (Dipped in 10% NH_4VO_3 Solution)

CWS Carbon - 1.9380 g.
NH_4NO_3 - 0.0629 g.
"H" - 0.7800 g. - H/C = 40.2%
H_2O - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	20	41.7	5.4
2	80	89.1	11.4
3	155	125.5	16.1
4	215	139.2	17.8
5	275	178.3	22.9
6	335	190.8	24.5
7	1450	340.5	43.7

TABLE V

DESORPTION OF MUSTARD FROM CARBON (Dipped in 10% $Hg(CN)_2$ Solution)

CWS Carbon - 1.713 g.
$Hg(CN)_2$ - 0.291 g.
"H" - 0.2625 g. - H/C = 15.3%
H_2O - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	45	10.6	4.0
2	120	17.0	6.4
3	180	19.1	7.3
4	240	23.9	9.1
5	300	26.3	10.0
6	360	31.5	12.0
7	1470	49.4	18.8

TABLE VI

DESORPTION OF MUSTARD FROM CARBON (Dipped in 10% $HgBr_2$ Solution)

CWS Carbon	-	1.596 g.
$HgBr_2$	-	0.404 g. - $HgBr_2$/C = 25.3%
"H"	-	0.2696 g. - H/C = 16.9%
H_2O	-	400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	30	5.3	1.9
2	60	6.7	2.5
3	120	10.1	3.7
4	180	10.8	4.0
5	240	14.3	5.3
6	300	17.6	6.5
7	360	19.9	7.4
8	1325	31.6	11.7
9	1385	32.9	12.2

TABLE VII

DESORPTION OF MUSTARD FROM ASC WHETLERITE

Whetlerite	-	2.0027 g.
Mustard	-	0.8440 g.
(H/C	=	42.1%)
Water	-	400 cc.

Sample No.	Time Min.	"H" Desorbed mg.	"H" Desorbed Wt. %
1	35	141.6	16.8
2	95	217.8	25.8
3	125	246.1	29.2
4	1141	375.9	44.5
5	1186	377.9	44.8
6	1291	385.6	45.7
7	1506	399.7	47.3

TABLE VIII

DESORPTION OF MUSTARD FROM CARBON (Dipped in $CuCO_3 \cdot Cu(OH_2)$ Solution
Basis: - 10% Cu present)

CWS Carbon - 1.8017 g.
Cu - 0.2013 g. - Cu/C = 11.15%
"H" - 0.3519 g. - H/C = 19.5%
Water - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	35	5.4	1.5
2	60	7.9	2.2
3	120	11.5	3.3
4	180	14.1	4.0
5	255	17.8	5.1
6	320	20.7	5.9
7	375	22.8	6.5
8	440	32.5	9.2
9	1655	54.3	15.5
10	1980	61.7	17.6

TABLE IX

DESORPTION OF MUSTARD FROM CARBON (Dipped in 10% $(NH_4)_2 MoO_4$ Solution)

CWS Carbon - 1.704 g.
$(NH_4)_2 MoO_4$ - 0.309 g.
"H" - 0.2615 g. - H/C = 15.4%
H_2O - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	35	4.3	1.6
2	65	5.6	2.1
3	140	9.5	3.6
4	200	13.7	5.2
5	260	13.9	5.3
6	420	15.0	5.7
7	1440	31.0	11.9
8	1500	29.4	11.2

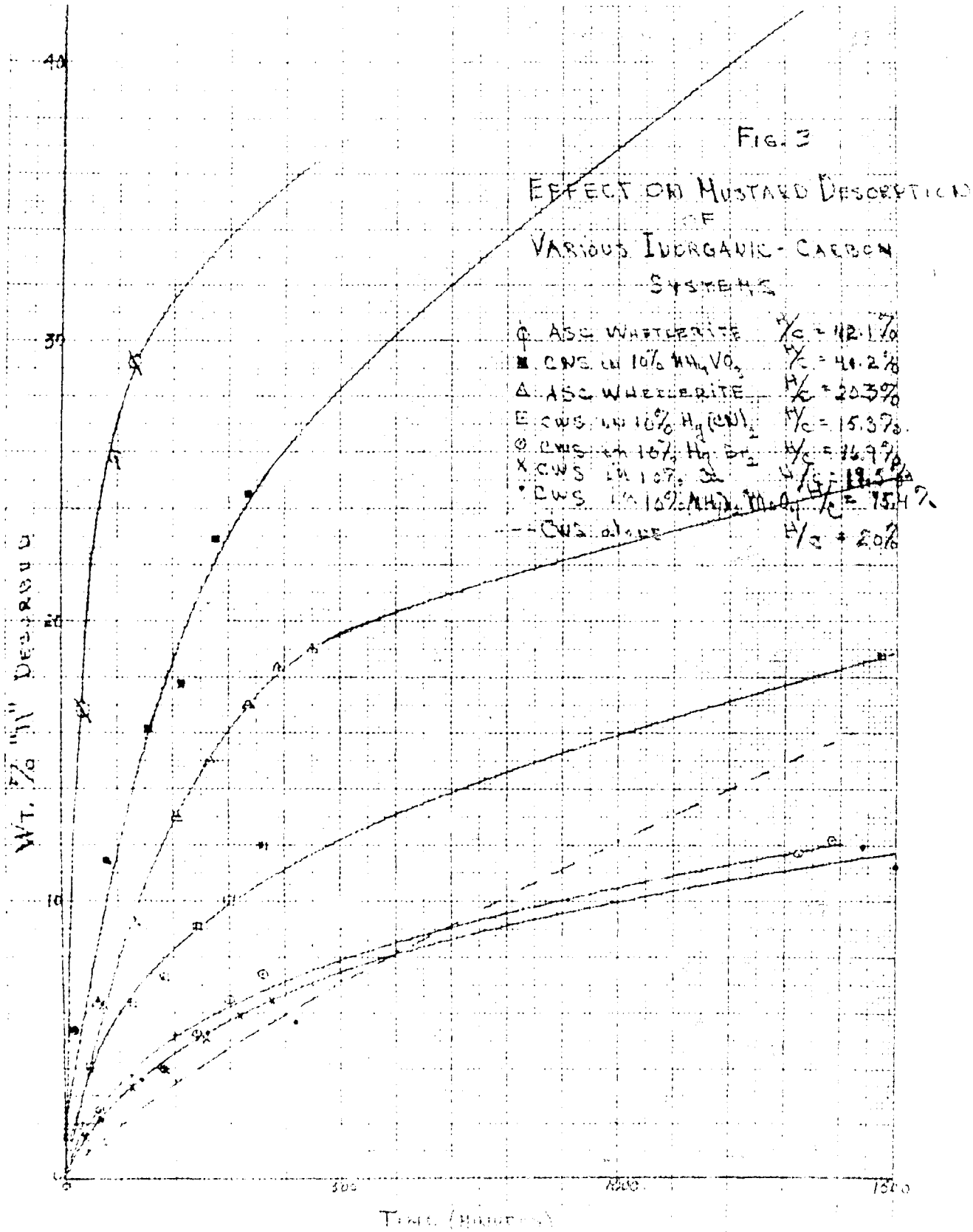


FIG. 3
EFFECT ON MUSTARD DESCRIPTION
OF
VARIOUS INORGANIC-CARBON
SYSTEMS
○ ASC WHITLOCKITE H/C = 12.1%
■ CWS IN 10% NH4VO3 H/C = 41.2%
△ ASC WHEELERITE H/C = 20.3%
E CWS IN 10% Hg(CN)2 H/C = 15.3%
⊙ CWS IN 10% HgBr2 H/C = 16.9%
X CWS IN 10% Se H/C = 19.5%
• CWS IN 10% (NH4)2MoO4 H/C = 15.4%
CWS ALONE H/C = 20%
WT. % "H" DESORBED
40
30
20
10
0
500
1000
1500
TIME (MINUTES)

TABLE X

DESORPTION OF MUSTARD FROM CARBON (Dipped in 10% Ethanolamine Solution)

CWS Carbon - 1.8500 g.
Ethanolamine - 0.1599 g.
"H" - 0.3476 g. - H/C = 18.8%
Water - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	15	6.95	2.0
2	45	11.9	3.4
3	75	17.2	4.95
4	130	25.9	7.45
5	208	32.4	9.3
6	280	39.6	11.4
7	340	46.4	13.4
8	400	49.8	14.3

TABLE XI

DESORPTION OF MUSTARD FROM CARBON (Dipped in 18% Sulfamic Acid Solution)

CWS Carbon - 1.7190 g.
Sulfamic Acid - 0.2889 g.
"H" - 0.3754 g. - H/C = 21.8%
Water - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	15	10.2	2.7
2	45	16.4	4.4
3	100	22.1	5.9
4	182	29.4	7.8
5	235	33.6	8.9
6	295	36.9	9.8
7	355	40.2	10.7
8	415	42.1	11.2

FIG. 9

EFFECT ON MUSTARD DESORPTION OF MISC. CARBON SYSTEMS

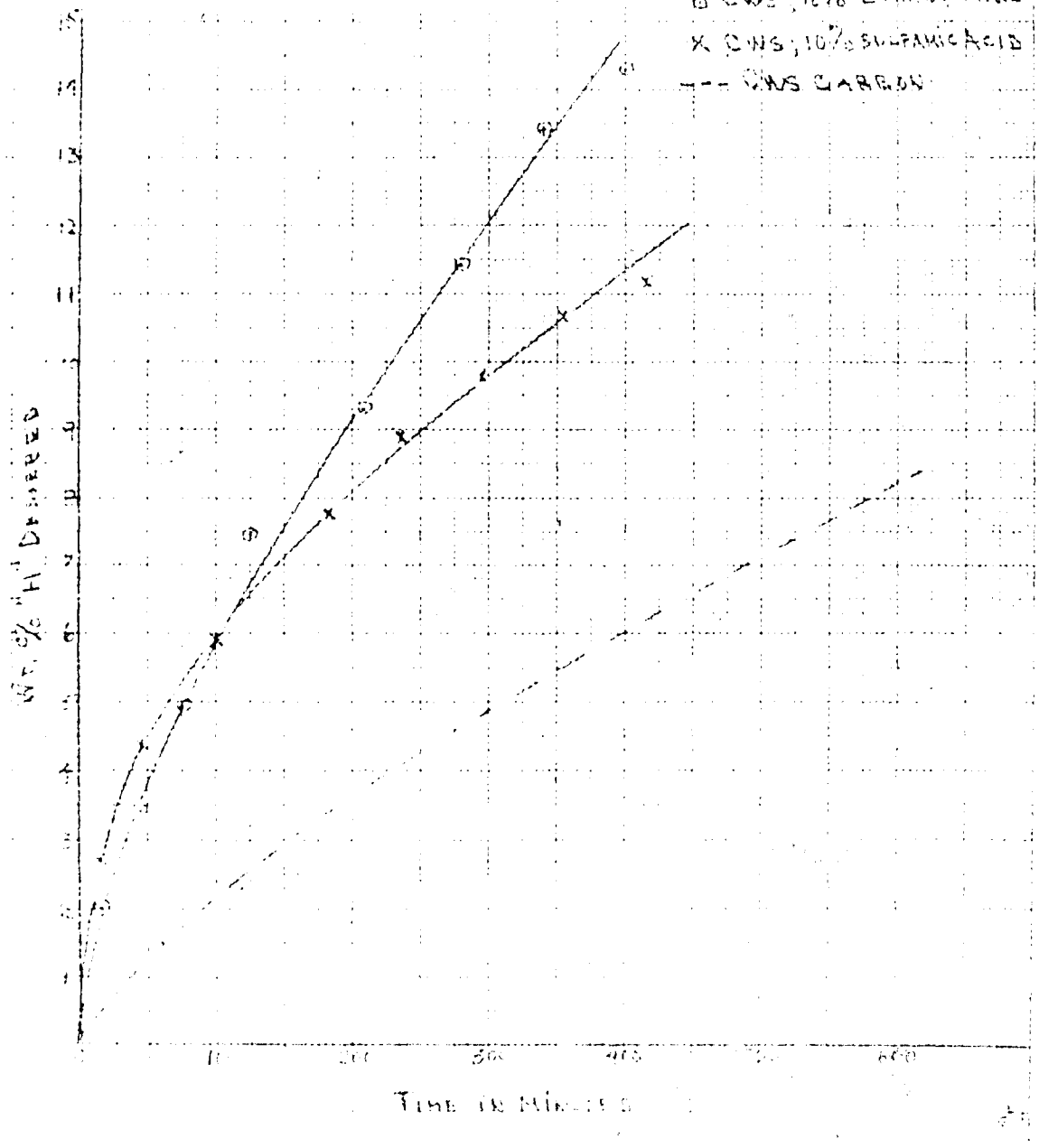


TABLE XII

DESORPTION OF MUSTARD FROM CARBON (Dipped in 10% H_2SO_4 Solution)

CWS Carbon - 1.8799 g.
H_2SO_4 - 0.1564 g. - H_2SO_4/C = 8.3%
"H" - 0.3719 g. - H/C = 19.8%
H_2O - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	60	18.3	4.9
2	120	23.1	6.2
3	240	28.2	7.6
4	300	27.7	7.5
5	465	34.7	9.3
6	575	36.4	9.8
7	1785	53.1	14.3

TABLE XIII

DESORPTION OF MUSTARD FROM CARBON (Dipped in 20% H_2SO_4 Solution)

CWS Carbon - 1.4723 g.
H_2SO_4 - 0.54 g. - H_2SO_4/C = 36.7%
"H" - 0.3280 g. - H/C = 22.3%
H_2O - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	30	-	-
2	60	14.9	4.6
3	92	14.6	4.5
4	160	16.0	4.88
5	1105	18.6	5.67
6	1185	19.5	5.97
7	1245	21.4	6.5
8	1555	20.9	6.4
9	1595	21.7	6.6

TABLE XIV

DESORPTION OF MUSTARD FROM CARBON (Dipped in 10% H_3PO_4 Solution)

CWS Carbon - 1.7300 g.
H_3PO_4 - 0.277 g. - H_3PO_4/C = 16.0%
"H" - 0.2633 g. - H/C = 15.2%

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	35	4.1	1.5
2	95	6.7	2.6
3	155	7.4	2.8
4	240	8.9	3.4
5	300	9.5	3.6
6	360	10.5	4.0
7	420	11.5	4.4
8	1435	13.8	5.3
9	1480	14.6	5.5

TABLE XV

DESORPTION OF MUSTARD FROM CARBON (Dipped in HCl Solution)

CWS Carbon - 1.8458 g.
HCl -
"H" - 0.3878 g. - H/C = 19.4%
H_2O - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	30	3.2	0.83
2	75	4.0	1.0
3	165	6.0	1.6
4	225	7.4	1.9
5	285	8.0	2.1
6	350	9.1	2.3
7	1350	23.3	6.0
8	1410	23.9	6.2
9	1515	24.6	6.4
10	1635	25.9	6.7

TABLE XVI

DESORPTION OF MUSTARD FROM CARBON (Dipped in HAc Solution)

CWS Carbon - 1.9995 g.
HAc - -
"H" - 0.3462 g. - H/C = 17.4%
H_2O - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	30	2.6	0.8
2	90	3.9	1.1
3	130	5.0	1.5
4	210	5.5	1.6
5	300	6.8	2.0
6	360	8.0	2.3
7	420	8.4	2.4
8	450	9.2	2.7
9	1440	21.1	6.1
10	1500	20.6	6.0
11	1515	20.8	6.02
12	1575	21.7	6.3
13	1640	22.7	6.6

TABLE XVII

DESORPTION OF MUSTARD FROM CARBON (Dipped in 10% HNO_3 Solution)

CWS Carbon - 2.0046 g.
HNO_3 - -
"H" - 0.3853 - H/C = 19.2%
H_2O - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	30	3.3	0.9
2	90	4.7	1.2
3	140	5.3	1.4
4	215	6.2	1.6
5	275	6.7	1.8
6	335	7.9	2.1
7	395	8.8	2.3
8	455	9.2	2.4
9	1435	13.5	3.5
10	1495	14.3	3.7
11	1555	14.3	3.8

Fig. 5

Effect on Mustard Desorption
of
Various Acid + Carbon Systems

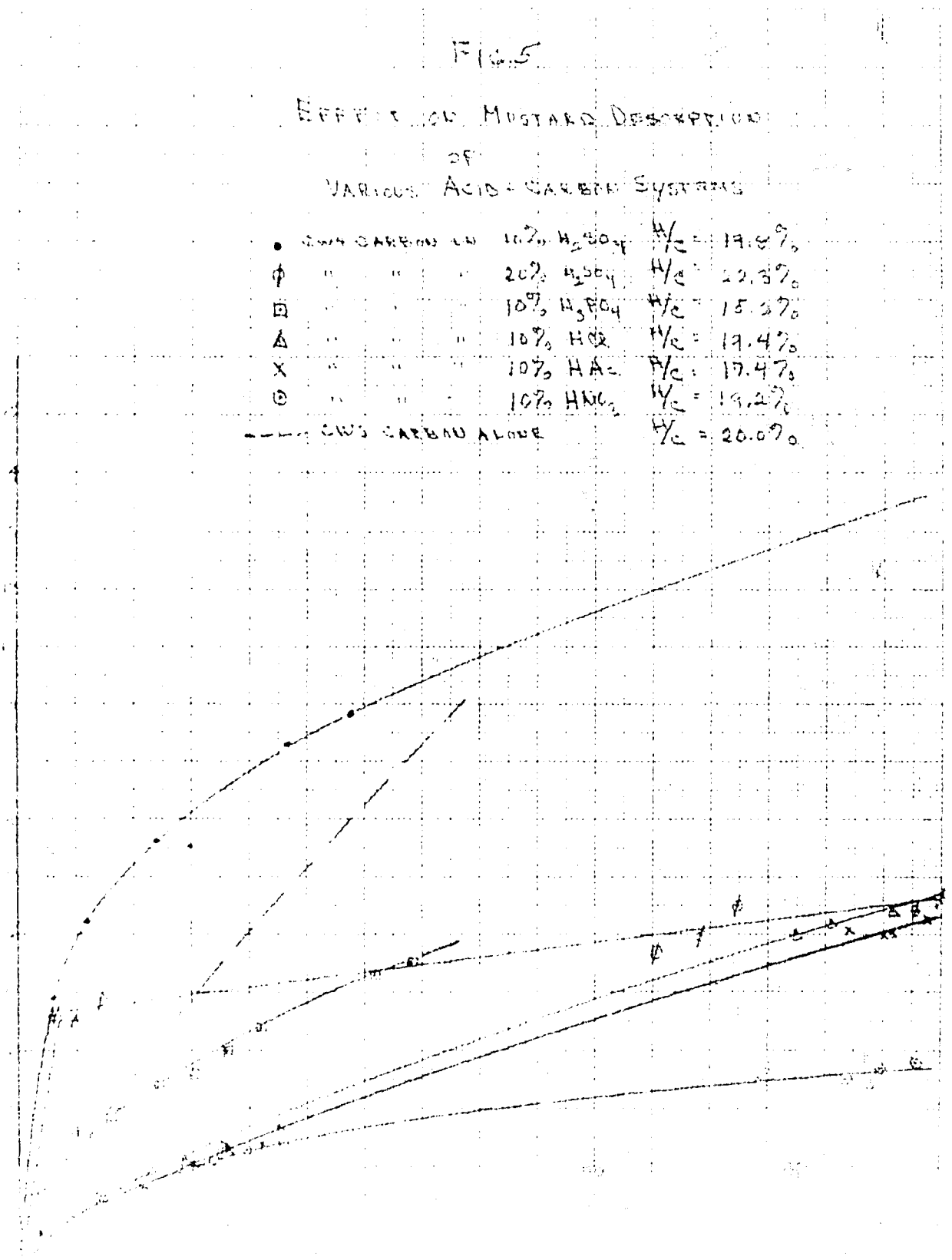


TABLE XVIII

DESORPTION OF MUSTARD FROM CARBON (Washed 3 times with conc. HCl)

CWS Carbon - 1.9992 g.
"H" - 0.3811 g. - H/C = 19.1%
H_2O - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	30	5.2	1.4
2	60	6.9	1.8
3	120	10.9	2.9
4	150	12.0	3.2
5	215	16.3	4.3
6	275	19.9	5.2
7	335	24.2	6.4
8	385	26.8	7.0
9	1335	51.9	13.6
10	1395	54.1	14.2
11	1455	60.4	15.9

TABLE XVIX

DESORPTION OF MUSTARD FROM CARBON (Dipped in 10% HCl Solution)

CWS Carbon - 2.0011 g.
"H" - 0.3684 g. - H/C = 18.4%
H_2O - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	60	4.1	1.1
2	90	6.1	1.7
3	120	8.4	2.3
4	180	9.7	2.6
5	240	12.2	3.3
6	300	15.6	4.2
7	1170	37.9	10.2
8	1230	41.3	11.2

TABLE XX

DESORPTION OF MUSTARD FROM CARBON (Dipped in 10% HCl Solution)

CWS Carbon - 2.0255 g.
"H" - 0.1932 g. - H/C = 9.6%
H_2O - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	21	0.6	0.3
2	60	1.4	0.7
3	180	1.8	1.0
4	240	2.7	1.4
5	360	3.95	2.1
6	1315	14.7	7.6
7	1365	15.1	7.8
8	1390	16.4	8.5

TABLE XXI

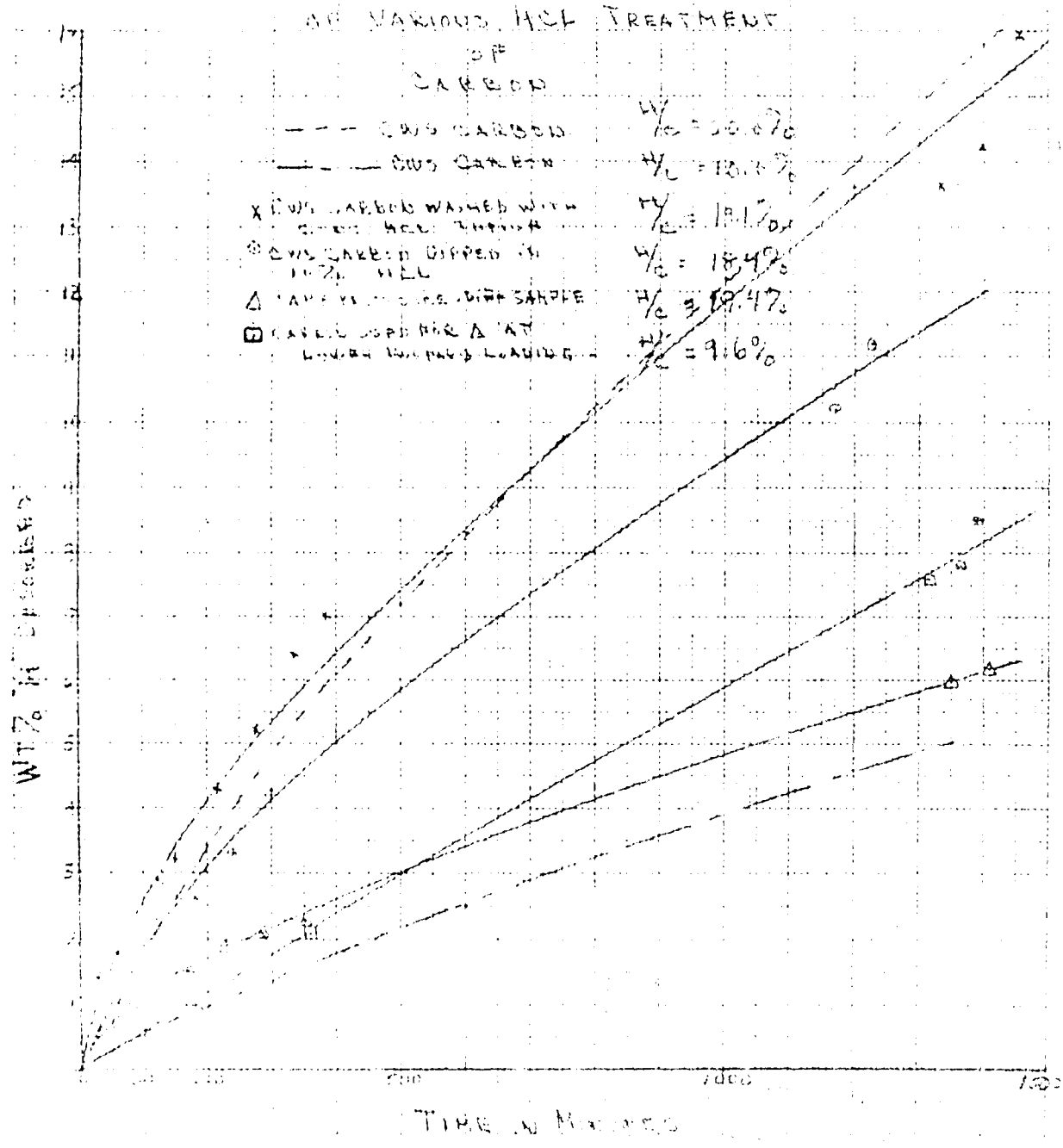
DESORPTION OF MUSTARD FROM CARBON (Dipped in 10% HNO_3 Solution)

CWS Carbon - 2.0023 g.
"H" - 0.2158 g. - H/C = 10.8%
H_2O - 400 cc.

Sample No.	Time Min.	"H" Desorbed Mg.	"H" Desorbed Wt. %
1	23	6.6	3.0
2	60	5.4	2.5
3	180	11.1	5.1
4	240	13.8	6.4
5	300	16.3	7.6
6	395	19.5	9.1
7	1420	39.3	18.3
8	1480	40.1	18.6
9	1535	39.8	18.5
10	1620	38.3	17.8

FIG. 6

EFFECT ON MUSTARD DECONTAMINATION OF VARIOUS HCl TREATMENT OF CARBON



DISCUSSION:

ASC Whetlerite:

As stated in the Abstract, comments on the data on ASC Whetlerite ageing this month can be only negative. While we have not proved that organic solvent contamination was the cause of the disconcerting results obtained, it certainly is a very likely possibility. Painting in the compressor room was in progress during all the equilibrations after the initial tests. Equilibration at 80% RH takes 44 to 48 hours at about 3.5 l./min. so around 10,000 liters of contaminated air was used. We have placed a large carbon absorber in the air line to avoid this in the future.

Mustard:

Water soluble organics such as ethanolamine, dimethylamine and pyridine probably increase the rate of mustard desorption in water by increasing the mustard solubility particularly at the water interface. The impregnation with materials such as ammonium vanadate and ammonium molybdate where the salt is decomposed to the oxide or hydrated oxide leaves solids which would either fill or plug inner pores. Their effect is probably more in this area than in changing the picture at the water mustard interface.

In the case of non-volatile acids such as sulfuric and phosphoric the acids on drying retreat to the inner pores, the last part to be dehydrated, and thus occupy the areas where desorption is slowest. Presumably the other carbon surface was benefited by acidification and the two effects balance.

Since CWS Carbon is exposed to air at relatively high temperatures after activation, one can presume the surface to be oxygenated. It is known that hydrogen treatment makes the surface more hydrophobic and CO_2 or O_2 more hydrophilic. While hydrogen ion would be expected to make the surface more hydrophilic it would probably have more tendency to bond with the negative Cl on mustard. At any rate, the effect of washing with volatile acids appears definite and while not sufficient to prevent desorption it is a lead. We expect to try ion exchange resins and CWS Carbons after hydrogen treatment in the near future.

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